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Modification of metal surface under the radiation exposure in the atomic-scale

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Abstract. Modification of the surface structure by radiation exposure in metallic materials was investigated by field ion microscopy. Irradiation regimes for the formation of amorphized and nanostructured states in the near-surface platinum regions were determined. Established modes of radiation processing of engineering alloys Pd50Cu30Ag20 to obtain in the near-surface volume structures like microduplex-type structure (the particle size of the phases to 100 nm) with the required combination of properties.

1. Introduction

One of the urgent problems of surface structure modification is to study the interaction of charged particle beams with the surface of the material in the near-surface volume (at a distance of ~1–100 nm from the irradiated surface). In fact, the creation of films or coatings in this size range of irradiation of materials.

A significant change in the atomic structure of the surface can be caused by phase-structural transformations that occur as a result of radiation exposure. By varying the parameters of radiation exposure (energy and fluence of irradiation), it is possible to obtain the necessary changes in the atomic crystal structure of solid solutions in the near-surface layers of a certain depth.

The method of field ion microscopy (FIM) was chosen for the diagnosis of such states. FIM capabilities allow us to study the near-surface volume of irradiated materials by controlled removal of atoms from the surface.

2. Experimental

The objects of interaction with charged Ar^+ beams were samples of structural alloy Pd50Cu30Ag20 (wt. %), pre-deformed by 70 %, annealed for 1 h at $T = 1123$ K (cooling in water) and platinum (purity 99.99 %). Samples intended for the study were prepared in the form of needle emitters with a radius of curvature of the top 30–50 nm by electrochemical polishing of wire blanks of materials. Then the samples were certified in a field ion microscope to provide an atomically smooth surface before irradiation.

Irradiation of Pt-tip samples certified in a field ion microscope was carried out by accelerated gas ion beams (Ar^+), fluences (F) – 10^{16} – 10^{18} ion/ cm^2 and ion current density $j=150$ ($T=70^\circ\text{C}$) and $200 \mu\text{A}/\text{cm}^2$ ($T=200^\circ\text{C}$), respectively.



Alloy Pd50Cu30Ag20 (wt. %) irradiated with $E = 20$ keV, at $j = 200\text{--}340$ $\mu\text{A}/\text{cm}^2$. This corresponded to a change in the temperature of the sample under irradiation within $T = 450\text{--}700$ K ($T_{\text{kr}} = 873$ K phase transformation to Pd50Cu30Ag20).

The bombardment was carried out in a direction parallel to the axis of the sample-tip. The irradiated tip samples were again placed in a field ion microscope and, by registering the field ion micro images of the surface with a photo or video camera with a controlled removal of atomic layers, the experimental material was obtained for the subsequent analysis of the modified structure.

3. Results and considerations

An atomically smooth surface of the Pt emitter tip was prepared for in situ FIM irradiation. Ion images of such samples showed almost perfect ring contrast of metal single crystals (Figure 1a), indicating the absence of structural defects.

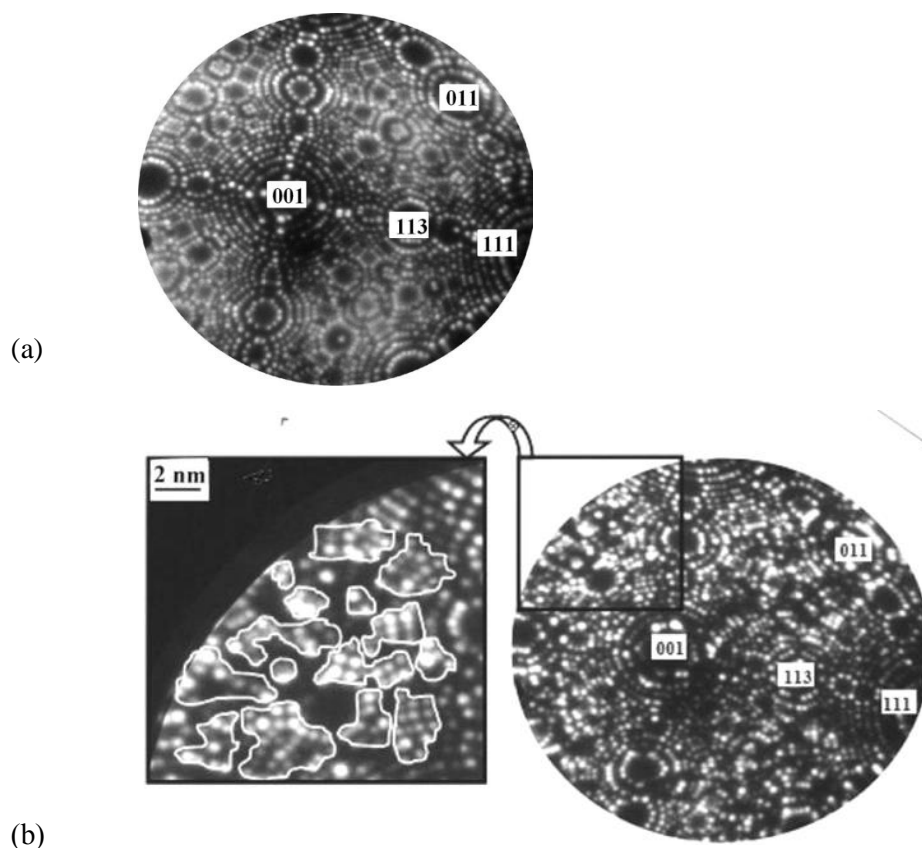


Figure 1. Neon images Pt: (a) – certified; (b) – irradiated, $F=10^{16}$ ion/ cm^2 .

After irradiation with fluence 10^{16} ion/ cm^2 , $j=100\text{--}150$ $\mu\text{A}/\text{cm}^2$ ($T=70$ C), Fig. 1b, and fluence 10^{17} ion/ cm^2 , $j=200$ $\mu\text{A}/\text{cm}^2$ ($T=200$ C), figure 2(a), the ionic contrast of platinum has changed. The presence of a ring pattern of contrast indicates that the phase state of the metal remains crystalline with such irradiation fluences. As a result of the analysis of the ring pattern of contrast after irradiation (figure 1(b)), were found violations in the ring the contrast of images of faces of the crystal. It was the violation in an annular pattern of ionic contrast register defectiveness of the crystal. The detected ion contrast indicates the presence of a nanoscale block structure in the near-surface volume of the material [1], (figure 1(b)). The ion contrast of the irradiated platinum compared to the contrast of the certified Pt was observed in a layer 1.5 nm deep from the irradiated surface.

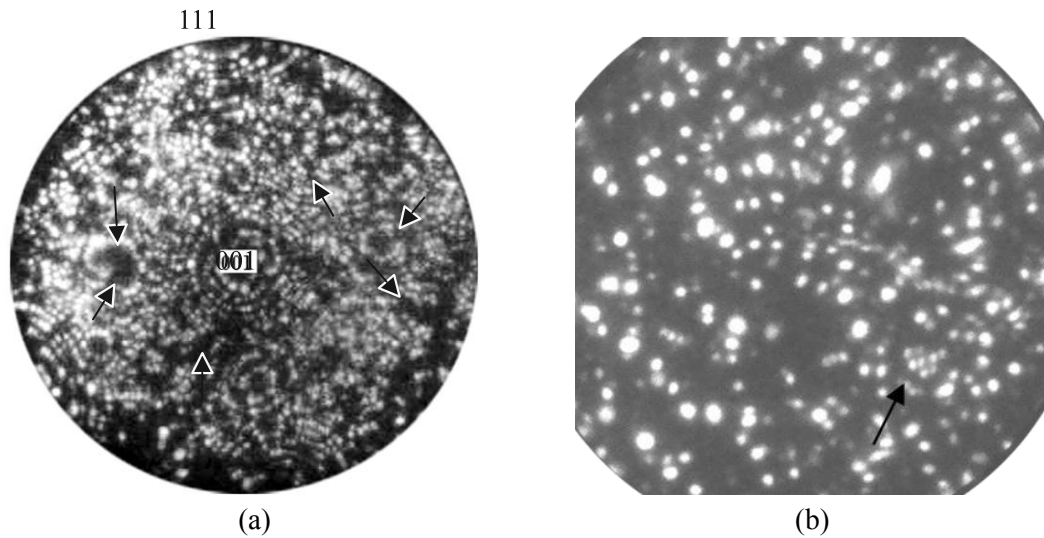


Figure 2. Neon images Pt: (a) – $F=10^{17}$ ion/sm², ($T=70$ C). A typical ionic contrast of the boundaries of nanoblock and defects is shown by arrows; (b) – $F=10^{18}$ ion/sm² ($T=200$ C), (the arrow marks the region of the crystalline state of the metal).

Analyzing the ionic contrast of the atoms in the nanoblocks, figure 1(b), it is obvious that atoms practically occupy their nodes in the crystal lattice of the material. The nanoblocks themselves are disoriented relative to each other.

Irradiation to higher fluence ($F=10^{17}$ ion/sm²), figure 2(a), gives the effect of forming a block nanocrystalline structure (with a block size of 1–5 nm). This structure is observed in the near-surface volume with a depth of not less than 20 nm from the irradiated surface. As a result, it was possible to determine the transverse and longitudinal dimensions of nanocrystalline blocks and the width of the boundary area between the nanoblocks. According to the authors' estimates, the width of the boundary region varied from 0.4 to 0.8 nm at various sites of nanoblock boundaries in irradiated platinum [2].

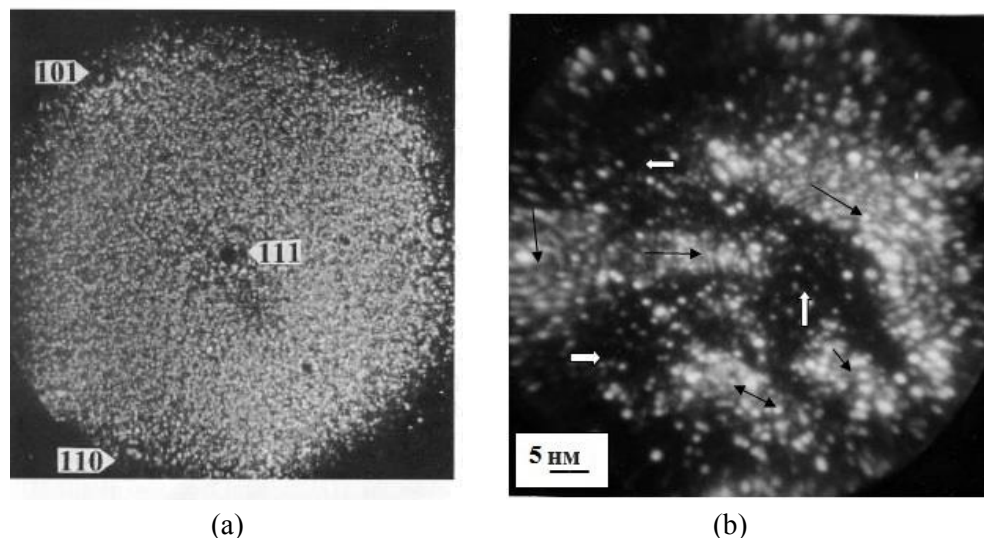


Figure 3. Neon image of a certified triple solid solution Pd50Cu30Ag20 (wt. %) (a); ion contrast of the micro-duplex structure obtained after irradiation of the surface Pd50Cu30Ag20 (wt.%) Ar^+ (b) with $F = 10^{18}$ ion/sm², at $j = 300$ $\mu\text{A}/\text{sm}^2$; black arrows show PdCu phase particles ordered by type B2, white arrows – AgPd phase particles (solid solution).

For figure 2(b) the ion image of the irradiated surface of platinum with a fluence of 10^{18} ion/cm² is given. Based on the contrast of the micro image of the atomic-pure surface of platinum, it is obvious that with an increase in the fluence to 10^{18} ion/cm², the phase state of the metal practically becomes amorphous. The proof is the structure less contrast of the arrangement of atoms in the near-surface layers. This contrast corresponds to the ionic contrast of amorphous materials obtained by ultra-fast cooling. According to the authors, the amorphization of pure metal (Pt) occurs in a near-surface volume 12 nm deep from the irradiated surface.

It is shown in [3] that high strength and plastic properties of PdCuAg alloys were obtained by forming the so-called micro-duplex structure in the material.

In [4] it was found that the size of the near-surface volume in which the structural phase transformation took place also changed depending on the value of the ion current density at other constant irradiation parameters. At a current density in the beam of 300 $\mu\text{A}/\text{cm}^2$, the phase transformation extended to a depth of ~ 100 nm from the irradiated surface, and at $j = 340 \mu\text{A}/\text{cm}^2$ – not less than 600 nm. It should be noted that at $j = 200 \mu\text{A}/\text{cm}^2$ no discontinuous decomposition occurs in the triple solid solution.

When changing the ion implantation modes (only the value of the ion current density varied), it was found that for $j = 300 \mu\text{A}/\text{cm}^2$ in a triple solid solution of PdCuAg, a micro-duplex structure arose (figure 3(b)).

4. Conclusion

1. Regimes were defined of radiation exposure for creating amorphized states in subsurface volume of platinum.

2. The analysis of surface modification, near-surface volume and boundary regions of nanoblock after irradiation with medium-energy ions of 10-30 keV is carried out.

3. The modes of radiation treatment of engineering alloys of Pd50Cu30Ag20 type are established, as a result, we can obtain a structure of the micro-duplex type (particle size of phases up to 100 nm) in the near-surface volume.

Acknowledgments

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